

Section 5.5 Title: Using and Maintaining the Solvent System

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P.I.: Prof. John F. Berry

Prior Approval: This procedure is NOT considered hazardous enough that prior approval is needed from the Principal Investigator.

Involves Use of Particularly Hazardous Substance (PHS)? No
 Carcinogen Reproductive Toxin High Acute Toxicity
Does this procedure require medical surveillance? No
Does this require use of a fit-tested respirator? No

Brief Description of Procedure:

Dispense dry solvent into an oven-dried flask from the solvent system.
Maintenance tasks for the solvent system.

Location: *List the locations (buildings/rooms) where this procedure may be performed. For use of a PHS indicate a more precise location within the room, if appropriate, as a designated area.*

Daniels Chemistry - 6325

Chemicals Involved:

Chemical	Physical or Health Hazard (e.g. carcinogen, corrosive)
Pentane	Flammable liquid

Other Hazards: *Include hazards, other than chemical, that may be present during operation of the procedure.*

Flammable solvent; asphyxiation (during refill)

Exposure Controls: *(Check all that apply)*

PPE: Safety Glasses Face Shield Chemical Splash Goggles
 Chemical Apron Gloves (Nitrile) Lab Coat
 Respirator (type) Other:

Engineering Controls:

Fume Hood Biosafety Cabinet Glove box
 Vented gas cabinet Other: Vent line to fume hood (during refill)

Administrative Controls: *List any specific work practices needed to perform this procedure (e.g., cannot be performed alone, must notify other staff members before beginning, etc.).*

Notify others when refilling. Do not refill alone. Receive training before use.

Task Hazard Control Table: *For procedures involving numerous steps, it may be convenient to indicate specific requirements for individual tasks in the table below:*

N/A

Waste Disposal: *Describe any chemical waste generated and the disposal method used.*

Dispose of excess solvent in the appropriate solvent carboy.

Accidental Spills: *Describe the procedure for handling small chemical spills that may occur during this procedure. Note that for large spills it may be appropriate to call 911.*

Small spills may be cleaned with an absorbing material. The material should be placed in a fume hood to dry after the spill has been cleaned.

Decontamination Procedures (required for PHS use): *Describe the procedure for decontamination of personnel and equipment.*

N/A

Training: *Describe any training needed prior to performing this procedure. Include training performed in-lab and any required demonstrations of competency.*

Training is required. Training is performed by the group member(s) responsible for this apparatus or another lab member they have approved. The procedure will be demonstrated at least once and new members will be supervised their first time.

Principle Investigator Approval: I have reviewed this procedure and approved it for use. Note: Modifications to the procedure may require update to this form.

Name: John F. Berry

Signature: _____

Date: _____

Using and Maintaining the Solvent System

Note: The Vacuum Atmosphere Solvent Systems are known to have a high failure rate. As of February 2019, only the system for pentane is able to effectively dry the solvent. The other systems were discarded when attempts to secure maintenance were unsuccessful. It is believed that the pumps employed in these systems degrade rapidly upon exposure to most organic solvents.

Solvent System Use:

1. Obtain a hot flask from the oven. Place it under vacuum or in a desiccator to cool.
2. Turn on the solvent system vacuum pump. It should run for at least 10 minutes prior to connecting your flask. Place your name card on the pressure gauge.
3. If the system does not have the appropriate dispensing nozzle, attach one with a pair of wrenches. The nozzle should point directly downward, be secured very tightly, and be capped with a clean Teflon sleeve.
4. Connect your cool flask to the dispensing nozzle. Do not grease the joint! Secure your flask to the dispensing nozzle with a rubber band and use a cork ring on a lab jack to support the weight.
5. Turn V1 to the left (vacuum). Turn V2 to the right, exposing your flask to vacuum.
6. Let the flask evacuate for at least 5 minutes.
7. Turn V1 to the right (inert gas) to fill your flask with N₂ for ~10 seconds.
8. Turn V1 back to the left and repeat steps 6 and 7 two additional times.
9. Turn V1 to the left for the fourth time. Allow it to evacuate for at least one minute.
10. Turn V2 downward (off). Turn V1 to the right (inert gas). Turn V2 to the left to dispense solvent.
11. Indicate on the solvent volume chart the size of the flask you are filling.
12. Once you have collected the desired amount of solvent, turn V2 to the right. Any solvent remaining in the nozzle will be pushed into your flask.
13. Turn V2 downward. Turn V1 downward.
14. With a greased ground glass stopper or rubber septum at the ready, disconnect your flask from the dispensing nozzle. Quickly seal your flask with the stopper or septum.
15. Quickly degas your solvent, as it was exposed to the atmosphere while disconnecting from the dispensing nozzle.
16. If you are done with the solvent system, turn off the vacuum, remove your name tag, and double check that you marked your solvent use volume.

Troubleshooting Tips:

1. When filling a flask near to full, the vacuum in the flask which pulls solvent from the reservoir may decrease to the point where solvent stops flowing. If this happens, turn V2 to the right to push out any remaining solvent and refill your flask with N₂. Turn V1 to the left for ~5 seconds to apply a quick vacuum. (Do not leave V1 open for more than 10 seconds, as this will pull your solvent through the vacuum pump.) Turn V2 to the left to dispense additional solvent. Turn V1 back to the right. Repeat this process as necessary.
2. After several liters of solvent have been dispensed from the reservoir, the pressure can decrease, slowing the rate of solvent dispensing. If you believe this is the case, you can

turn the Sparge Gas valve on (~10 seconds). If this does not remedy the issue, the reservoir should be refilled.

Tasks for individuals responsible for solvent system maintenance:

Solvent refills:

1. The solvent reservoir holds 4 liters of solvent, but may need to be refilled after only 3 have been dispensed. At least 48 hours prior to refilling, ensure that a 4L solvent bottle is available and treated with activated molecular sieves. Pre-drying solvent helps prolong the lifetime of the drying column of the solvent system.
2. Refilling the solvent system can release a large volume of solvent vapor into the atmosphere. Ensure that no unnecessary personnel are nearby and that someone in the lab knows you are refilling the solvent system. Refill the system quickly.
3. Bring the pre-dried solvent to the solvent system, as well as a 1L Erlenmeyer flask and a large glass funnel.
4. Place the narrow end of the sparge line in the nearby fume hood and the wide end near the solvent system.
5. Turn the Sparge Gas valve on. Immediately open the sparge vent at the top of the reservoir. Do not allow the reservoir to build pressure.
6. Unscrew the wing nut securing the sparge vent to the reservoir. Place the sparge vent and o-ring seal on the table.
7. Pour 1L of solvent into the Erlenmeyer flask. Use the funnel to carefully pour the solvent into the reservoir via the opening where the sparge vent connects.
8. Continue pouring solvent via the Erlenmeyer until the reservoir is full (solvent will begin to splash out).
9. Replace the o-ring seal and sparge vent. Tighten the wing nut firmly.
10. Connect the sparge line to the sparge vent. Allow the solvent to sparge for 30 minutes.
11. Indicate in the solvent system logbook the date and volume of solvent filled.
12. After the solvent has sparged, turn the sparge vent off. Immediately turn off the Sparge Gas valve. Do not allow the reservoir to build pressure.
13. Coil and replace the sparge line. Replace the solvent use form on the solvent system body.

Replacing the solvent column:

1. The drying columns are rated to remove ~48 grams of H₂O. This should handle over 250 liters of pre-dried solvent.
2. When the drying column needs to be replaced (as determined by Karl Fischer titration), the reservoir should be drained through normal use. Do not refill the reservoir.
3. Follow the manufacturer's instructions for replacing the drying cartridge.
4. Removing the joints from the solvent system can be difficult. The machine shop can use a vice to open the joints.
5. The used molecular sieves from the drying column should be dried completely in a glass container or Rubbermaid tub. Placing them directly in a solid waste jug will melt the jug.
6. The new molecular sieves are pre-activated. One package should refill the column fully. Any excess sieves should be discarded.

7. When you change the drying column, also replace the filter on the dispensing spout. Follow the manufacturer's instructions.